Magnetic order and crystal structure study of YNi₄Si-type NdNi₄Si

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A B S T R A C T
Magnetic measurements and neutron powder diffraction investigation of the magnetic structure of the orthorhombic YNi₄Si-type (space group Cmmm) NdNi₄Si compound are presented. The magnetocaloric effect of NdNi₄Si is calculated in terms of the isothermal magnetic entropy change and it reaches the maximum value of −3.3 J/kg K for a field change of 50 kOe near Tc = 12 K. Below ~ 12 K, NdNi₄Si exhibits a commensurate b-axis collinear ferromagnetic ordering with the Cmmm magnetic space group in a zero magnetic field. At 1.5 K, the neodymium atoms have the magnetic moment of 2.37(5) μB. The orthorhombic crystal structure and its thermal evolution are discussed in comparison with the CaCu₅-type compound.

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1. Introduction

Recently, the orthorhombic derivative of the CaCu₅-type, namely the YNi₄Si-type (space group Cmmm), RNi₄Si compounds (R = Y, La, Ce, Sm, Gd–Ho) were reported [1]. These compounds supplement the known series of the CaCu₅-type RNi₅ compounds and RNi₄Si solid solutions [2,3]. The orthorhombic distortion of parent CaCu₅-type compounds may be considered a prospective route for optimizing their magnetic and hydrogen storage properties [4–6]. In order to test its feasibility, it requires a systematic investigation of the magnetic properties of YNi₄Si-type RNi₅, which can be in comparison with those of the well-known CaCu₅-type RNi₅ and RNi₄Si compounds. The early reports of the magnetic properties and structures of CaCu₅-type RNi₅ and RNi₄Si [7–11] and YNi₄Si-type RNi₅ [1,12,13] (R = Gd, Tb, Dy) permit us to draw the following preliminary conclusions. In the case of [Tb, Dy]Ni₄Si the ferromagnetic ordering temperature increases from the CaCu₅-type RNi₅ across CaCu₅-type RNi₄Si to YNi₄Si-type RNi₅, whereas the Curie point decreases from GdNi₅ to the CaCu₅-type GdNi₄Si and the Curie temperatures of CaCu₅-type and YNi₄Si-type GdNi₄Si are the same. We suggest that the transformation of their magnetic properties in the orthorhombic distortion of CaCu₅-type lattice results from the initial ab-plane ferromagnetic-like ordering of Tb and Dy sublattices and possible c-collinear ferromagnetic ordering of Gd sublattice. The Ni ions have little to no magnetic moments in these compounds.

The initial CaCu₅-type NdNi₅ shows ferromagnetic ordering below 7.2 K with isothermal magnetic entropy change of ~8.45 J/kg K for a field change of 0–50 kOe [14,15]. The substitution of Si for Ni in the NdNi₅Si solid solution leads to increase of Curie point from 7.2 to 9.2 K and decrease of isothermal magnetic entropy change from ~ 8.45 J/kg K to ~7.3 J/kg K for a field change of 0–50 kOe [16]. This work aims to understand the effects of orthorhombic distortion from the parent CaCu₅-type NdNi₅ to the YNi₄Si-type NdNi₄Si compound through the magnetic measurements and neutron powder diffraction.

2. Material and methods

The NdNi₅Si sample was prepared by arc melting of the stoichiometric amounts of Nd (99.9 wt%), Ni (99.95 wt%) and Si (99.99 wt%). The sample was annealed at 1070 K for 200 h in an
argon-filled and sealed quartz tube and subsequently quenched in ice-cold water. The structure, purity and composition of the polycrystalline sample were evaluated using powder X-ray diffraction (XRD) and electron microprobe analysis. The XRD data were obtained on a Rigaku D/MAX-2500 diffractometer (CuKα radiation, 2θ = 10–80°, step 0.02°, 1 s/step). An INCA-Energy-350 X-ray EDS spectrometer (Oxford Instruments) on a Jeol JSM-6480LV scanning electron microscope (20 kV accelerating voltage, beam current 0.7 nA and beam diameter 50 μm) was employed to perform the microprobe analyses of the sample. Signals from three points were averaged and estimated standard deviations were 1 at% for Nd (measured by L-series lines), 1 at% for Ni and 1 at % for Si (measured by K-series lines).

DC magnetization of the polycrystalline NdNi₄Si sample was measured on a commercial Physical Property Measurement System (Quantum Design PPMS-DynaCool) in the temperatures range of 5–300 K with an applied field of 5 kOe in the zero-field-cooled (ZFC) and field-cooled (FC) modes. The isothermal saturation magnetization was measured for the magnetic field change from 0 to 50 kOe at various temperatures.

Neutron diffraction experiments were carried out at the high flux reactor of the Institut Laue Langevin (Grenoble, France). The data were collected in a zero magnetic field on the two-axis D1B powder diffractometer equipped with a 1300 cell curved detector spanning the 2θ range of 130° [17]. The temperature ranges were 128–30 K with a step of ~10 K and 25–1.5 K with a step of ~5 K. The neutron wavelength of 2.5238 Å was selected by the (002) reflection of a pyrolytic graphite monochromator and the 2θ step was 0.1°.

### 3. Theory/calculation

The unit cell data were derived from the powder XRD using the Rietan program [18,19] in the isotropic approximation at room temperature. The paramagnetic susceptibility was fitted to the Curie–Weiss law to obtain the effective magnetic moment and paramagnetic Curie temperature [20]. Magnetocalaroric effect (MCE) was calculated in terms of the isothermal magnetic entropy change, ΔSm, using the magnetization vs. field data obtained during the phase transition and employing the thermodynamic Maxwell equations [21]. The neutron diffraction data were refined with the FULLPROF program [22]. The magnetic space groups [23,24] were used for the analysis of neutron diffraction data.

### 4. Results

#### 4.1 Crystal structure

Both the microprobe and X-ray powder analyses show that NdNi₄Si is a single-phase sample. The microprobe analysis yielded the Nd₁₇(1)Ni₆₈(1)Si₁₅(1) composition, and the X-ray powder analysis

### Table 1

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<th>Site</th>
<th>Atom</th>
<th>x/a</th>
<th>y/b</th>
<th>z/c</th>
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* Cmmm = {1, m, m, m, T, 2z, 2z, 2z} × {1, 1/2, 1/2, 0} = Pmmm × {1, 1/1, 1/2, 0}.
* Cmcm = {1, m, m, m, T, 2z, 2z, 2z} × {1, 1/2, 1/2, 0} = Pmmm × {1, 1/1, 1/2, 0}.
* Pmmm = {1, m, m, m, T, 2z, 2z, 2z}.
* Pmm2 = {1, m, m, m, 2z, 2z}.
* Pmmm = {1, m, m, m, T, 2z, 2z, 2z} × {1, 1/2, 1/2, 0} = Pmm m × {1, 1/1, 1/2, 0}.
* Pmm2 = {1, m, m, m, 2z, 2z}.
* Pmmm = {1, m, m, m, T, 2z, 2z, 2z} × {1, 1/2, 1/2, 0} = Pmm m × {1, 1/1, 1/2, 0}.
* Pmm2 = {1, m, m, m, 2z, 2z}.
* Pmmm = {1, m, m, m, T, 2z, 2z, 2z} × {1, 1/2, 1/2, 0} = Pmm m × {1, 1/1, 1/2, 0}.
* Pmm2 = {1, m, m, m, 2z, 2z}.

Fig. 1. (a) Magnetization and inverse magnetic susceptibility of NdNi₄Si as a function of temperature in 5 kOe and (b) magnetization vs. magnetic field of NdNi₄Si at 2 K.
confirmed the YNi4Si-type structure with the Cmmm space group. The lattice parameters were refined as a = 0.51354(2) nm, b = 0.83006(3) nm, c = 0.39707(2) nm, V = 0.16926(4) nm³, b(312)a = 0.93320(3), and the atomic sites Nd (2a) [0, 0, 0], Ni1 (4i) [0, 0.3424(5), 0], Ni2 (4f) [1/4, 1/4, 1/2] and Si (2c) [0, 1/2, 1/2] (R = 4.5%). The atomic positions for the Nd 2a, Ni1 4i and Ni2 4f sites in the Cmmm space group with the corresponding symmetry operators are given in Table 1.

4.2. Magnetic properties and magnetocaloric effect

The zero-field-cooled (ZFC) and field-cooled (FC) magnetization data recorded during heating in 5 kOe are shown in Fig. 1a. The FC data are indicative of a typical ferromagnet, while the ZFC data suggest presence of weak competing antiferromagnetic interactions, which can be easily overcome in small magnetic fields. The paramagnetic susceptibility of NdNi4Si follows the Curie-Weiss law in the temperature range 90–300 K (inset in Fig. 1a). The fit to the Curie-Weiss law yielded a paramagnetic Weiss temperatures θp = −21(5) K and the effective moment per formula unit Meff = 3.8 μB/μ. The negative θp can be seen as the development of antiferromagnetic-type interactions, which is also observed in the ferromagnetic SmNi4Si compound [25]. The refined Meff is close to the theoretical magnetic moment of Nd3+ (3.62 μB) [26], indicating Nd is trivalent in the compound. However, the neutron diffraction studies do not confirm the presence of magnetic moments on Ni, and thus only Nd atoms are assumed to carry localized magnetic moments. In this case, the effective magnetic moment of the Nd atoms is 3.8 μB, and a slight increase of ~0.2 μB over the theoretical value can be attributed to the polarization of conduction electrons, predominantly the Nd 5d ones, through the 4f–5d exchange interactions.

The magnetization vs. magnetic field for NdNi4Si at 2 K is plotted in Fig. 1b. A rapid increase in the magnetization at low fields is typical for a ferromagnet and is attributed to the domain growth. However, a subsequent slow linear increase and non-saturating behavior is indicative of the competing antiferromagnetic interactions and/or strong anisotropy. A strong magnetic anisotropy is likely to be present in the NdNi4Si structure and does not allow the magnetic moments to be fully oriented in the magnetic field. The saturation magnetic moment reaches the value of 1.4 μB/Nd in 50 kOe, which is significantly smaller than the theoretical saturation moment 3.27 μB of Nd3+ [26]). NdNi4Si shows the hysteresis at 2 K with residual magnetization Mr = 0.8 μB/μ and coercive field Hc = 3.4 kOe (Fig. 1b). Existence of significant coercivity in the hysteresis cycle may be an indication of the presence of uniaxial magnetic anisotropy in the NdNi4Si compound, which is verified as a ferromagnetic ordering along the b axis through the neutron diffraction studies below.

The magnetocaloric effect of NdNi4Si in terms of the isothermal magnetic entropy change, ΔSm, was calculated from the saturation magnetization data (Fig. 2a). A numerical integration is performed using the following formula: ΔS(T)mag = Σ(Mi+1 - Mi)/Ti+1 - TiΔH, where ΔH is a magnetic field step and Mi and Mi+1 are the values of magnetization at temperatures Ti and Ti+1, respectively [21]. The magnetic entropy change, ΔSm, for ΔH = 0–50 kOe is plotted in Fig. 2b. As expected for a second order magnetic transition, ΔSm peaks around the Curie temperature and has a maximum value of ~3.3 J/(kg K).

4.3. Magnetic structure

Above 10 K, the neutron diffraction patterns of NdNi4Si in a zero applied field correspond to the paramagnetic state, and at T = 10 K a set of commensurate magnetic reflections with a Kf = [0, 0, 0] wave vector indicates the magnetic ordering of NdNi4Si (Fig. 3). The ordering temperature found from the neutron diffraction study is in good agreement with the value deduced from the magnetization measurements of TC ~ 12 K (Figs. 1a and 4a).

Fig. 2. (a) Magnetization vs. magnetic field and (b) the isothermal entropy change, −ΔSm, of NdNi4Si around the magnetic transition.

Fig. 3. Neutron diffraction patterns of NdNi4Si (a) at 25 K (paramagnetic state) and (b) at 1.5 K (b-axis ferromagnet with Kf = [0, 0, 0] wave vector. The first row of ticks refers to the nuclear Bragg peaks whereas the second row of lines refers to the magnetic reflections. The (hkl) of strongest magnetic reflections are indicated in (b).
A commensurate $b$-axis collinear ferromagnetic model of NdNi$_4$Si fits best with the NDP data (Fig. 5). Within this model, the calculated magnetic moments for the Ni$_1$ and Ni$_2$ sublattices are close to or within the error bar with $M_{Ni}/C240.13(15)\mu_B$, which means that the significance of such ordered magnetic moment on Ni cannot be confirmed. The other tested commensurate variants yielded no magnetic ordering for the Ni sublattices either. The negligible presence of the Ni magnetic moments in this compound results most probably from the Ni-3$d$ band filling via electronic hybridization with the Nd and Ni neighbor states. The $b$-collinear magnetic ordering of the Nd sublattice corresponds to the $Cmm\bar{m}$ magnetic space group as shown in Table 1. The neodymium magnetic moment reaches $2.37(5)\mu_B$ at 1.5 K, a value less than the theoretical value of $3.27\mu_B$ expected for Nd magnetic moment [26] (Fig. 4b and Table 2), which is often observed in some rare-earth-based intermetallic compounds [27,28].

The unit cell of NdNi$_4$Si undergoes anisotropic distortion down to the ferromagnetic transition temperature: the cell parameters decrease with $a_T/a_{298} < b_T/b_{298} < c_T/c_{298}$, and below the ferromagnetic ordering the cell parameters remain almost constant (Fig. 4c and Table 2). As the NdNi$_4$Si structure is an orthorhombically distorted variant of the hexagonal CaCu$_5$ structure, the $b_T/a_{298}$ ratio can be used to estimate the degree of the distortion and its progression with temperature. The $b_T/a_{298}$ ratio stays almost constant from 298 K to 1.5 K and it is far from the unit value that corresponds to the transformation from the orthorhombic YNi$_4$Si-type lattice to the hexagonal CaCu$_5$-type lattice.

5. Discussion

The saturation magnetization at 2 K and in 50 kOe yielded a magnetic moment of 1.1 $\mu_B$ per neodymium atom (Fig. 1b), whereas neutron diffraction studies at 1.5 K and in a zero applied field indicated a complete ferromagnetic ordering of NdNi$_4$Si with $2.37\mu_B$/Nd (Fig. 4b). It can be understood that a large magnetic anisotropy prevents a parallel alignment of all the Nd moments in the polycrystalline sample with the magnetic field even at 50 kOe.

Falkowski et al. [16] reported the magnetic properties of CaCu$_5$-type NdNi$_4$Si compound unfortunately without providing crystal data. Based on the aforementioned structural and magnetic data, we propose that transformation from the initial CaCu$_5$- to the YNi$_4$Si-type NdNi$_4$Si unit cell (compression along the $b$-orthorhombic axis as in TbNi$_4$Si [12]) leads to the modification in the Nd environment and thus the changes in their magnetic properties, such as, increasing of temperature of magnetic ordering from 9.2 K up to 12 K, reorientation the neodymium moments in the $ab$-plane normal to the compression of unit cell, decreasing of magnetocaloric effect from $-7.3\text{J/kg K}$ of CaCu$_5$-type NdNi$_4$Si down to $-3.3\text{J/kg K}$ of YNi$_4$Si-type NdNi$_4$Si in field of 0–50 kOe as in TbNi$_4$Si [12]. Such a transformation leads to the appearance of distinct hysteresis loop at 2 K of the YNi$_4$Si-type NdNi$_4$Si in contrast with the CaCu$_5$-type NdNi$_4$Si (Table 3).

6. Conclusions

The new YNi$_4$Si-type NdNi$_4$Si supplements the series of YNi$_4$Si-type RNi$_4$Si compounds with $R=Y$, La, Ce, Sm, Gd–Ho. Compared to the CaCu$_5$-type NdNi$_4$Si compound, the YNi$_4$Si-type counterpart has the relatively high ferromagnetic ordering temperature (9.2 K vs. 12 K), the small magnetocaloric effect ($-7.3\text{J/kg K}$ vs. $-3.3\text{J/kg K}$ for $\Delta H=50\text{kOe}$), and the large magnetic anisotropy at low...
temperatures. This work suggests that such an orthorhombic distortion from the initial CaCu₅-type unit cell can take place in the other YNi₄Si-type solid solutions, e.g., PrNi₄Si. The orthorhombic distortion may be used as a prospective route for optimization of permanent magnetic properties in the family of CaCu₅-type rare earth materials.

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References

[16] < www.ill.eu >, Yellow Book.

Table 2
Crystallographic and magnetic parameters of YNi₄Si-type NdNi₄Si at different temperatures: cell parameters a, b and c, unit cell volume V, b(3[1/2]a), the atomic position of Nd1 atom yNd1, M eff the magnetic moments of Nd along the b-axis with K0=[0, 0, 0] wave vector. Reliability factors are: R<sub>r</sub> for the crystal structure and R<sub>r</sub> for the magnetic structure.

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<th>T (K)</th>
<th>Cell parameters (nm)</th>
<th>V (nm&lt;sup&gt;3&lt;/sup&gt;)</th>
<th>b(3[1/2]a)</th>
<th>yNd1 (%)</th>
<th>Atom</th>
<th>M&lt;sub&gt;eff&lt;/sub&gt; (μB)</th>
<th>R&lt;sub&gt;r&lt;/sub&gt; (%)</th>
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<td>298&lt;sup&gt;b&lt;/sup&gt;</td>
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<td>4.5</td>
<td>Nd&lt;sup&gt;1&lt;/sup&gt;, Nd&lt;sup&gt;2&lt;/sup&gt;</td>
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* Atomic sites of YNi₄Si-type NdNi₄Si (space group Cmmn): Nd (2a) [0, 0, 0], Nd (1d) [0, 0, 3/4], Nd (4f) [1/4, 1/4, 1/2] and Si (2c) [0, 1/2, 1/2].
* X-ray data.

Table 3
Magnetic properties of CaCu₅-type NdNi₄, NdNi₄Si and YNi₄Si-type NdNi₄Si compounds.

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<th>H&lt;sub&gt; coerc&lt;/sub&gt; (kOe)</th>
<th>ΔS&lt;sub&gt;mag&lt;/sub&gt; (J/kg K)</th>
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<td>(4.2 K, 90 kOe)</td>
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<td>1.1</td>
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* This work.


